Puff by puff determination of pH of an aqueous solution of mainstream cigarette smoke using a routine linear analytical smoking machine

1 Scope

This procedure specifies the method for trapping a single puff of mainstream cigarette smoke in degassed 0.1 N KCl solution and the subsequent pH analysis of the aqueous solution. The puff of mainstream cigarette smoke is generated and collected using a linear analytical smoking machine.

2 Normative references

At the time this procedure was written, the editions indicated were valid.

ISO 3308:1991. Routine analytical cigarette - smoking machine - Definitions and standard conditions.

ISO 3402:1991. Tobacco and tobacco products - Atmosphere for conditioning and testing.

ISO 7210:1997. Routine analytical cigarette - smoking machine - Additional test methods.

Federal Register, Volume 32, No.147, p. 11178, August 1, 1967.

Federal Register, Volume 45, No. 134, p. 46483, July 10, 1980.

Regulations to implement Texas health and safety code, "Disclosure of Ingredients in Cigarette and Tobacco Products."

3 Definitions

For the purpose of this procedure, the following definitions apply.

3.1 pH: A measure of the acidity or basicity of an aqueous solution measured as the negative logarithm of the activity of hydrogen ions.

3.2 Total particulate matter: That portion of the mainstream smoke which is trapped in the smoke trap, expressed as milligrams per cigarette (mg/cigt.).

3.3 Smoking process: The use of a smoking machine to smoke cigarettes from lighting to final puff..

3.4 Laboratory sample: The sample intended for laboratory inspection or testing and which is representative of the gross sample or the sub-period sample.

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3.5 Conditioning sample: The cigarettes selected from the test sample for conditioning prior to tests for total particulate matter yield.

3.6 Test portion: A group of cigarettes prepared for a single determination and which is a random sample from the test sample or conditioned sample as appropriate.

3.7 Conditioned sample: Conditioned cigarettes for total particulate matter yield tests.

4 Principle

Sampling of the test cigarettes. Conditioning of the test cigarettes. Smoking of test cigarettes on a linear analytical smoking machine equipped with a trap containing degassed 0.1 N KCl solution with the collection of a single puff of mainstream smoke at various specified intervals during the smoking process. Determination of pH of the aqueous solution.

5 Apparatus and reagents

Normal laboratory apparatus and reagents and in particular the following items:

5.1 Smoke trap apparatus. Manufactured by Research Glass, Richmond, VA, part number pm030496. The apparatus consists of a single glass trap, (referred to as an impinger). The impinger consists of two pieces; the trap body which has a 24/40 ground glass joint and a trap insert. The impinger trap insert is a glass sampling tube with a ground glass fitting at the neck for a tight connection to the trap body. The top of the impinger trap insert is equipped with a 12/5 socket joint on one end and a hose connection on the other end. The volume of the impinger is approximately 90 mL. The cigarette is inserted into a rubber cigarette holder that is mounted on a 0.5 inch diameter, medium wall glass tube, (referred to as the impinger inlet tube). This glass inlet tube has a 90° bend at a distance of 125 mm from the rubber cigarette holder, (this allows placement of the apparatus within the smoking machine hood behind the Filtrona 435 smoking bar / ashtray assembly with the glass inlet tube passing through the smoking bar). A glass ball joint is used to attach the glass inlet tube to the impinger. An approximately two inch section of ¼ inch I.D. polyvinyl chloride (PVC) tubing is attached to the exit end of the impinger. The other end of the PVC tubing is inserted, to a 9 mm insertion length, into the harmonized filter pad holder that is mounted onto the carrier eccentrics, carrier slide, and PVC tubing assembly that is reassembled behind the smoking bar.

NOTE 1 A drawing of the smoking trap apparatus is given in Figure 1.

NOTE 2 All glassware is washed prior to each use with a laboratory soap solution, (Alconox or equivalent), and dried at 100°C for a minimum of 3 hours.

5.2 Filtrona 435 smoking machine. Modified to comply with the requirements of ISO 3308. The filter carrier slide is removed from the smoking bar for the port(s) utilized for the smoking portion of this procedure.

NOTE 3 In order to trap a single puff of mainstream smoke two ports of the smoking machine are utilized for a single determination (one port is used to capture the specified puff and the other to divert the preceding puffs). Ports 4 and 5 were utilized within this procedure.

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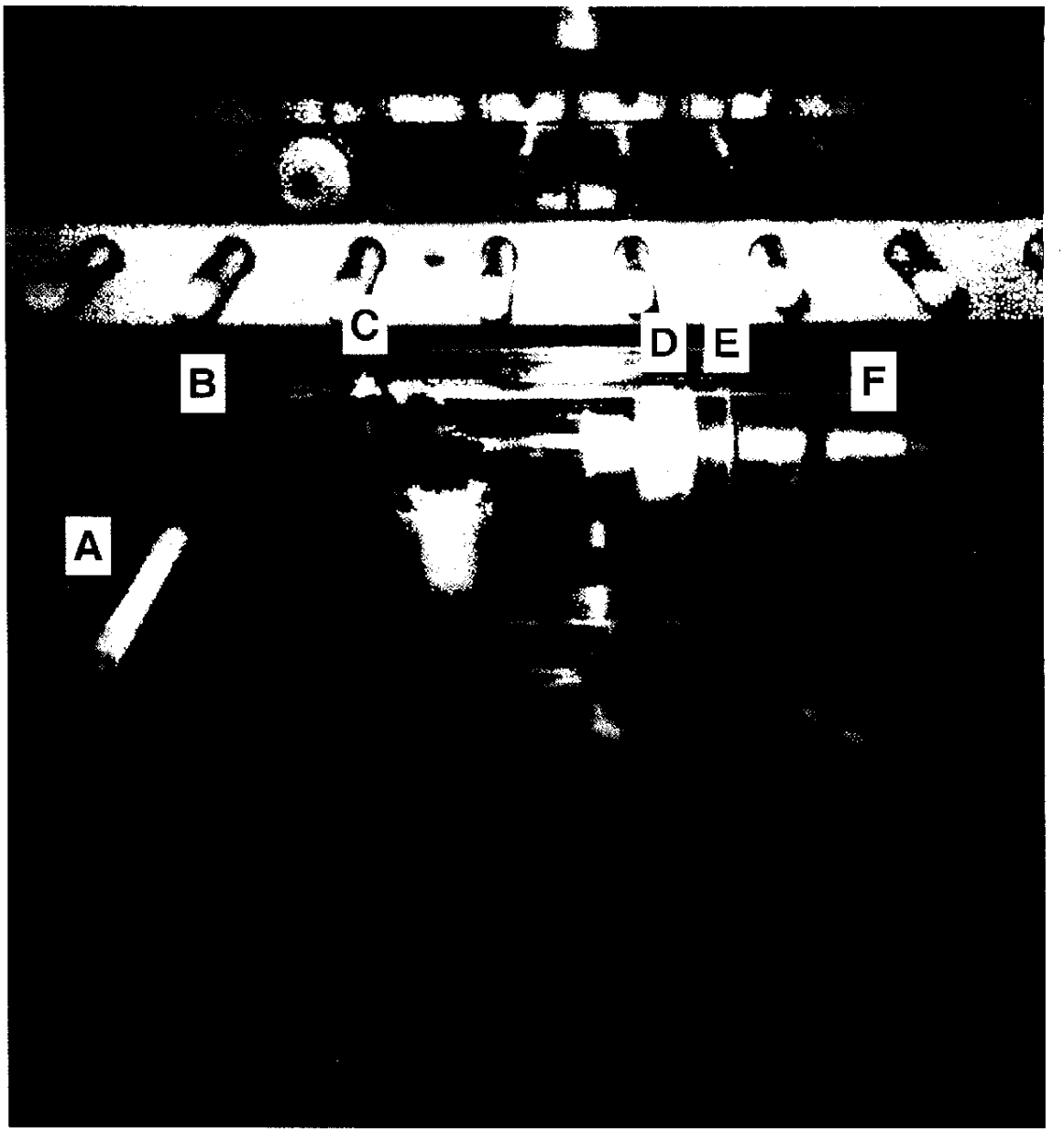

Figure 1 - Picture of the pH smoke trap apparatus. Where: A is the rubber cigarette holder; B is the impinger inlet tube; C is the glass ball joint with clamp; D is the harmonized glass fiber filter pad holder with PVC tubing inserted; E is the Filtrona carrier eccentric; and, F is the Filtrona carrier slide.

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Note 4 Due to the positioning of the smoke trap apparatus, (5.1), within the smoking machine hood, a maximum of four ports (two per determination) of the twenty available smoking machine ports may be utilized with this procedure. No criteria exist for which ports are utilized except for smoke trap spacing.

5.3 44 mm glass fiber filter pads. Manufactured by Whatman International Ltd. and certified to comply with the requirements of ISO 3308.

5.4 Filtrona harmonized filter pad holders. Manufactured by Filtrona to comply with the requirements of ISO 3308.

5.5 Omnidirectional air velocity probe and meter. Manufactured by Schiltknecht.

5.6 Soap bubble flow meter. Manufactured by Borgwaldt, graduated at 40 to 80 mL with a resolution of 0.2 mL.

5.7 Ruler. Certified for measurements to the nearest 0.5 mm.

5.8 pH meter. Model 25 Accumet pH/ion meter manufactured by Fisher Scientific.

5.9 Glass body combination electrode with Ag/AgCl internal reference element. Manufactured by Fisher Scientific.

5.10 50 mL Optifix solvent dispenser. Manufactured by EM Science. A calibration check of the dispenser is performed each shift, (to an accuracy of 50.0 ± 1.0 mL), using a certified 100 mL graduated cylinder.

5.11 Degassed 0.1 N KCl. 0.1 N KC1 is prepared using 7.4 g of certified ACS grade KCl per liter of HPLC grade water (available from Fisher Scientific). Prior to use, gas chromatography grade helium gas is bubbled, at a slow continuous stream, through the KCl solution for a minimum of 4 hours.

6 Sampling and sample preparation

6.1 Sampling

Remove one cigarette from each pack of the laboratory sample to form the conditioning sample.

6.2 Cigarette marking

The cigarette butt length is determined following FTC protocol as the overwrap, (tipping paper), length plus 3 millimeters. The 9 mm insertion and determined butt length are marked on each cigarette using a soft felt tip pen to avoid damage to the cigarette.

NOTE 5 Butt length is defined in ISO 3308 as the length of unburnt cigarette remaining at the moment when smoking is stopped.

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NOTE 6 If the determination of butt length has been performed on the laboratory sample as part of the total particulate matter determination, (ISO 4387), this determination does not need to be repeated on this test sample.

6.3 Cigarette taping

One-half of the cigarette overwrap is covered with Number 600, Scotch Brand tape. The width of the tape used is one-half the known circumference of the cigarette brand. The tape is applied lengthwise over the cigarette overwrap starting at the cigarette rod end and ending flush with the mouth end of the cigarette. Any tape extending past the end of the cigarette is cut off using a razor blade.

NOTE 7 For cigarettes of 24.8 to 25.2 mm circumference, the theoretical width of applied Scotch tape should be 12.4 to 12.6 mm. Commercially available, one-half inch (12.7 mm) Scotch tape is used to tape these cigarettes. The 12.7 mm commercially available tape is also used for cigarettes of 23.8 to 24.2 mm circumference. In addition, for cigarettes of 22.8 to 23.2 mm circumference, the tape is cut to an 11.7 mm tape width; and for cigarettes of 16.8 to 17.2 mm circumference, the tape is cut to an 8.7 mm tape width.

The actual coverage of the overwrap for cigarettes of these circumferences is thus slightly greater than one-half. The intent of this process is to cover one-half of the cigarette ventilation holes and this variance in tape width reflects ventilation hole coverage of slightly more than one-half.

6.4 Sample equilibration

The cigarettes are placed in an equilibration tray. This conditioning sample is equilibrated in a conditioning environment specified in ISO 3402 for a minimum of 48 hours and a maximum of 10 days.

7 Standard smoking conditions

The standard smoking conditions used are in accordance with ISO 3308:1991. 4, except for the following deviations specified per 105 CMR 660.000.

7.1 Puff volume

The standard puff volume measured in series with a pressure drop of 1 kPa is 45.0 mL ± 1.0 mL, (see Note 8). In order to achieve a puff volume of 45.0 mL at the entrance end of the smoke trap apparatus, the smoking machine is set at a puff volume of approximately 48.0 mL as measured from the harmonized filter pad holder as described in ISO 4387:1991. 7.5.3.3.

NOTE 8 ISO 4387:1991. 7.5.3.3 states that puff volume be set to 35.0 mL ± 0.3 mL. Due to the larger puff volume utilized in this procedure and the variation associated with measuring the puff volume through an aqueous solution a puff volume range of ± 1.0 mL was applied.

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7.2 Puff frequency

The standard puff frequency is one puff every 30 seconds with a standard deviation of not greater than 0.5 seconds.

8 Smoking process

8.1 Laboratory environment

The testing atmosphere in the laboratory where the smoking is carried out is in accordance with ISO 3402.

8.2 Smoking plan

The test portion is comprised of one cigarette randomly selected from the conditioning sample which comprises one determination of one puff collected at any selected point during the smoking process. Individual cigarettes are used for each puff collected from a test sample. For example, ten individual cigarettes are utilized to collect puffs 1 through 10 of a test sample.

8.3 Smoking process

The smoking machine set up is in accordance with ISO 4387 except for those changes noted in clauses 5.1 and 5.2 and section 7. Port 4 is set up as a conventional smoking port. Port 5 is equipped with the smoke trap apparatus. Fifty mL of degassed 0.1 N KCl solution is dispensed, (see clause 5.10), into the impinger trap. The impinger is secured with a 24/40 ground glass fitting clamp. The smoke trap apparatus is assembled per clause 5.1 for collection of the first puff. For collection of all subsequent puffs, the filter pad holder is placed on the carrier eccentric but is not attached to either the impinger or the carrier slide. All glass ball joints are secured with clamps. String is applied to the puff termination device on the smoking machine per ISO 4387 for those ports being utilized. For collection of the first puff, the test cigarette is inserted to the insertion mark into the rubber cigarette holder on port 5. The cigarette is lit and the puff is collected in the pH smoke trap apparatus. To collect subsequent puffs, a test cigarette is inserted to the insertion mark into the filter pad holder on port 4. The cigarette is lit and smoked on port 4, until ready to capture the designated puff. At that time, the carrier eccentric (which is attached to the rear of the filter pad holder) is then attached to the carrier slide and the front of the filter pad holder is attached to the exit end of the impinger trap. The lit cigarette is removed from port 4 and placed into the rubber cigarette holder on port 5 and the puff is collected in the pH smoke trap apparatus. After collection of the designated puff, the string is broken to terminate the smoking process and a timer is activated to track the time for the pH determination.

NOTE 9 Transfer of the lit cigarette and the assembly of the smoke trap apparatus must take place during the 30 second puff interval.

The smoke trap apparatus is disassembled. The impinger insert is removed from the impinger trap body. A magnetic stirring bar is placed in the impinger trap body containing the aqueous solution. The activated timer and the impinger trap body containing the aqueous solution so collected are utilized for the pH determination.

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9 pH determination

9.1 Daily pH calibration

Calibration of the pH meter is performed at the beginning of each shift. Calibration is performed as a two point standardization using pH 4 and pH 10 buffers. The slope or efficiency must be within 95% to 105% before proceeding.

9.2 Calibration standard check

A calibration standard check is performed using a pH 7 buffer before each test sample measurement. The buffer response must be 7.00 ± 0.10 pH units or recalibration is required.

9.3 pH determination of test samples

The impinger trap body containing the so collected aqueous solution and magnetic stirring bar from clause 8.3 is placed on a magnetic stirrer. The impinger trap body is held in place using a ring stand and clamp. At or before 10 minutes after the puff is collected, as indicated by the activated timer, the pH electrode is submerged into the test solution. At 15 ± 0.5 minutes after the puff is collected, the pH value is recorded.

NOTE 10 The pH meter is interfaced to a personal computer which allows automatic transmission of data from the pH meter into an Excel spreadsheet utilizing an Excel macro.

NOTE 11 All pH measurements are performed at 23.9 °C ± 2 °C and 60% ± 5% relative humidity.

10 Test report

The sample data is reported to 0.1 pH units.

The appropriate information concerning the characteristic data about the cigarette, sampling, and description of test is recorded in accordance with ISO 4387.

11 Repeatability and reproducibility

A total of seven samples with FTC tar levels varying from 2 mg to 22 mg were analyzed. Each sample was tested three times by one operator. The pH results for the seven samples were found to be not statistically different from one another.

Therefore, the difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value (r=0.14) on average not more than once in 20 cases in the normal and correct operation of the method.

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A collaborative study involving a suitable number of laboratories has not been performed for the determination of the method reproducibility (R) value.

12 Revision History

12.1 April 1998

Original procedure written by C. H. Callicutt and J. M. Garman.

12.2 September 1998

Document revised by C. H. Callicutt - Revisions include: (1) Addition of method repeatability values.

12.3 August 1999

Document revised by C. H. Callicutt – Revisions include: (1) Changed the minimum hours required to degas the 0.1 N KCl solution in 5.11. (2) Removed the reference to measuring cigarettes for butt length in 6.2. (3) Changed the puff volume range in 7.1.

No interlaboratory validation process has been conducted using this analytical method.

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